

Virginia Division of Consolidated Laboratory Services- Richmond, VA

Nitrogen (Ammonia or TKN) by Titrimetric, Following Distillation SM 4500-NH3 B+C-1997 (2011)					
<i>ADDITIONAL QC REQUIREMENTS FOR THIS METHOD: Certified or Accredited laboratories using this method are assessed to applicable requirements of SM 1020 and SM 4020. Also refer to appropriate checklist for TKN sample digestion.</i>					
Facility Name: _____ VELAP ID: _____					
Assessor Name: _____ Analyst Name: _____ Inspection Date: _____					
Relevant Aspect of Standards	Method Reference	Y	N	N/A	Comments
Records Examined: SOP Number/ Revision/ Date _____ Analyst: _____					
Sample ID: _____ Date of Sample Preparation: _____ Date of Analysis: _____					
1) Are samples for ammonia or TKN analysis refrigerated at $\leq 6^{\circ}\text{C}$, preserved to pH <2 with H_2SO_4 , and analyzed within 28 days?	40 CFR 136.3 Table 11				
2) Are samples checked for residual chlorine, and if present, were they treated with a dechlorinating agent?	4500-NH3-A.2				
3) Are all samples distilled prior to titration, or does the laboratory have acceptable comparability data on representative effluent samples to show that this preliminary distillation step is not necessary? <i>40CFR136 allows distillation by EPA 350.1 Rev. 2.0, SM4500-NH3B-1997(2011), or AOAC 973.49.</i>	4500-NH ₃ C.1, 40 CFR 136.3 Table 1B Footnote 6				
Distillation:					
4) Is 500-mL dechlorinated sample, or for samples with < 100 $\mu\text{g/L}$ ammonia/TKN, is 1000-mL sample used?	4500-NH ₃ B 4.b				
5) Is 25-mL Boric Acid added and pH adjusted to 9.5 with 6N NaOH using a pH meter?	4500-NH ₃ B 4.b				
6) Is distillation rate 6 to 10 mL/min with tip of the delivery tube below the surface of 50-mL boric acid solution?	4500-NH ₃ B 4.c				
7) Is at least 200-mL collected and then diluted to 500-mL with distilled water?	4500-NH ₃ B 4.c				
8) For sludge or sediment samples, is a wet portion weighed quickly to within $\pm 1\%$, equivalent to approximately 1 g dry weight?	4500-NH ₃ C.4.b				
9) For sludge or sediment samples, is the weighed portion washed into a distillation flask, diluted to 250 mL, and a piece of paraffin wax added to flask?	4500-NH ₃ C.4.b				
10) For sludge or sediment samples, are the samples then distilled as above, and 100 mL distillate collected for titration?	4500-NH ₃ C.4.b				
Notes/Comments: _____ _____ _____					

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Final Determination:					
11) Is ammonia-free water used in making all reagents and dilutions?	4500-NH ₃ C.3				
12) Are mixed indicator solution and indicating boric acid solution prepared at least monthly?	4500-NH ₃ C.3				
13) Is the appropriate sample volume selected for titration? 5-10 mg/L level use 250 mL; 10-20 mg/L use 100 mL; 20-50 mg/L use 50.0 mL; 50-100 mg/L use 25.0 mL	4500-NH ₃ C.1				
14) Is 0.02N H ₂ SO ₄ titrant standardized to 0.0200N per SM2320B.3c, or is 0.0200N titrant purchased? <i>For greatest accuracy, standardize titrant against an amount of Na₂CO₃ that has been incorporated in the indicating boric acid solution to reproduce the actual conditions of sample titration; 1.00 mL = 14 x normality x 1000 µg N. (For 0.02N, 1.00 mL = 280 µg N.)</i>	4500-NH ₃ C.3.c				
15) Are samples titrated with 0.02N H ₂ SO ₄ titrant until indicator turns pale lavender?	4500-NH ₃ C.4.c				
16) Is a blank carried through all steps of the procedure, and is the necessary correction applied to the results?	4500-NH ₃ C.4.d				
Notes/Comments:					